

**CONTINUOUS PROCESS FOR CONTROLLED  
EVAPORATION OF BLACK LIQUOR**

**Field of the Invention**

The present invention relates generally to continuous pulp production. More particularly, the invention relates to a method for evaporating weak black liquor to form strong black liquor.

**Background of the Invention**

In the pulping process, it is found that by separating spent pulping liquor (known as "weak black liquor") from the fibrous plant material, the purity of the pulp is improved. Weak black liquor typically contains 15 wt % dissolved and suspended solids of which about 80% are organic compounds and the remainder are inorganic compounds. The weak black liquor is separated from the pulp at a certain point in the process of pulp production. The weak black liquor is then subjected to evaporation to remove water. The weak black liquor, after it is concentrated, is then called "strong black liquor." The strong black liquor is then passed to a recovery boiler where the strong black liquor is combusted to recover inorganics, such as sulfur and sodium, for reuse in the pulping process. The steam that is produced by the combustion of the strong black liquor is used for heating in the pulping process.

The solids content in the strong black liquor must lie in a desired range to ensure proper burning in the recovery boiler. If too much water is evaporated, viscous strong black liquor may plug the spray nozzles or simply spray improperly, possibly extinguishing the burner flame. Conversely, if too little water is evaporated from the weak black liquor, the organics content will be too low to sustain a flame. Either case is dangerous, because the unburned strong black liquor would then fall into a pool of molten inorganic chemicals, which lies at the bottom of the recovery boiler, possibly causing an explosion. System interlocks are relied upon to shut off the flow of strong black liquor to the burner nozzles if the flame goes out.

Processes for the evaporation of weak black liquor to form strong black liquor are known in the art, such as direct contact evaporators, indirectly-heated concentrators, and multi-effect evaporators. However, the need still exists for a process that can control the concentration of strong black liquor reliably, consistently and precisely.

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### **Summary of the Invention**

The present inventive process provides a controlled method for evaporating weak black liquor to form strong black liquor. Advantageously, the method offers fast and precise control of viscosity of the strong black liquor. The control scheme allows for regulation of the concentration of the outgoing product to tight tolerances by manipulating the system pressure and the temperature of the incoming black liquor. It allows for good control over the product percent solids, and the product viscosity. Precisely controlling the viscosity and solids content of the strong black liquor sent to the recovery boiler would not only improve safety, but would also allow optimization of the operation of the recovery boiler, with respect to both chemical recovery capacity and energy efficiency.

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The inventive process is a process for concentrating weak black liquor to form strong black liquor, said process comprising: a) preconcentrating said weak black liquor; b) heating said preconcentrated weak black liquor in a heating zone under sufficient pressure to prevent said preconcentrated weak black liquor from boiling in said heating zone; c) passing said preconcentrated weak black liquor having a vapor pressure and having at least one volatile component, into an evaporation zone through one or more orifices; d) applying pressure in said evaporation zone that is lower than the vapor pressure of said preconcentrated weak black liquor as it is passed into said evaporation zone, which allows for flash evaporation of at least a portion of said at least one volatile component of said preconcentrated weak black liquor from said preconcentrated weak black liquor to form strong black liquor; e) adjusting temperature in said heating zone, pressure in said heating zone and pressure in said evaporation zone to allow for evaporation of an amount of said at least one volatile component from said preconcentrated weak black liquor to form said strong black liquor; and f) collecting said strong black liquor from said evaporation zone.

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### **Brief Description of the Drawings**

Figure 1 is a process flow diagram of an embodiment of the present inventive method; and

Figure 2 is a graph showing evaporator response time for sugar solution, with the normalized change in % solids versus the time, in minutes, for such a change in pressure (two changes) and temperature (two changes).

### **Detailed Description of the Invention**

An embodiment of the present invention is illustrated in Figure 1 by a process flow diagram. In this embodiment, weak black liquor from the wood pulping and washing steps of a pulp production process is preconcentrated in a preconcentrator **12**. The bulk of the water, aqueous media or solvent media in the weak black liquor is removed in this step.

The preconcentrated weak black liquor is then pumped, using a pump **14**, to a heating zone **16** where it is heated. A temperature sensor **18** monitors or senses the temperature of the heating zone **16**. The preconcentrated weak black liquor is kept under sufficient temperature and pressure in the heating zone **16** to keep the preconcentrated weak black liquor from boiling there.

There is a pressure sensor **20** and another temperature sensor **22** in the system prior to an evaporation zone (a "zone" is a chamber, enclosed area, etc.) **24**, which are used to sense the pressure and the temperature of the preconcentrated weak black liquor just prior to the preconcentrated weak black liquor entering the evaporation zone **24**. The preconcentrated weak black liquor is then passed into the evaporation zone **24** through an inlet **26** and a die **28** comprising one or more orifices. The die **28** is constructed such that the pressure drop through the die should prevent the preconcentrated weak black liquor from boiling until it has nearly entered, or has actually entered the evaporation zone. In order for the flash evaporation of the preconcentrated weak black liquor to take place in the evaporation zone **24**, pressure is controlled in the evaporation zone **24** such that it is lower than the vapor pressure of the heated preconcentrated weak black liquor as it enters

the evaporation zone **24**. The strong black liquor is cooled as it passes through the evaporation zone **24** and then goes through an outlet **30**.

A vapor exit port **34** is attached to the evaporation zone **24** where the vapor that was evaporated out of the strong black liquor in the evaporation zone **24** is removed from the evaporation zone **24**. Another pressure sensor **36** is located outside of the vapor exit port **34** or could be somewhere connected to the evaporation zone **24** itself, and is used to sense the pressure in the evaporation chamber **24** or just outside of it. In order for the vapor to exit the evaporation zone **24** the pressure needs to be lower outside of the evaporation zone **24** than inside. The lower pressure outside of the evaporation zone **24** may be provided by a vacuum system **40** that is connected to the system via a condenser **38** where most of the vapor is condensed for removal. Alternatively, vapors may pass directly to the pressure control or the vacuum system.

The strong black liquor is collected in a collection vessel **42**. It is then pumped out of the system by a pump **44**. A conditions sensor **46** senses, measure or infers such things as viscosity, pressure, concentration and heating value of the strong black liquor and is found at or near the exit for the strong black liquor or is attached to the collection vessel. These measurements or inferences can be used in controlling the operation of the system.

### **Preconcentrating**

The preconcentrating step may be performed using a preconcentrator **12**. Apparatuses that may be used as preconcentrators include, for example, multi-effect evaporators.

“Preconcentrating” means removing a portion of at least one volatile component in a solution or liquid medium prior to the first step of a process.

The volatile component of the weak black liquor that is removed or evaporated is mostly or all water. Other ingredients in the weak black liquor may also be evaporated, although that is not most desirable.

### Heating

The heating step of the present inventive method is performed in a heating zone **16**. The heating is preferably done using a heat exchanger. The heating step may be performed, however, by any suitable heating means or device. Some examples of other heating means include, but are not limited to, direct steam injection and microwaves.

The preconcentrated weak black liquor is heated to a temperature in the heating zone that is suitable so that the preconcentrated weak black liquor will have a temperature that is above the boiling point of the preconcentrated weak black liquor at the pressure in the evaporation zone.

The pressure in the heating zone is maintained so that the preconcentrated weak black liquor is not allowed to boil. The range of pressures depends upon the composition of the preconcentrated weak black liquor and the temperature of the heating zone and of the preconcentrated weak black liquor.

Appropriate temperature and pressure may be calculated using standard thermodynamic relationships. Methods for doing these calculations can be found in references such as R.C. Reid et al., *The Properties of Gases and Liquids*, 4<sup>th</sup> Ed., McGraw-Hill, New York, NY, 1987, Chapters 7 and 8, and Appendix A.

### Evaporation

After being heated in the heating zone **16**, the preconcentrated weak black liquor is passed through an inlet or conduit and through a die **28** or nozzle or some sort of orifice into the evaporation zone itself. The die or nozzle may be of any suitable shape and configuration that allow the heated preconcentrated weak black liquor to pass through into the evaporation zone **24**. The shape and/or number of orifice(s) through which it passes may allow for increase in the surface area of the preconcentrated weak black liquor that is exposed in the evaporation zone as it is passed through the zone. The preconcentrated weak black liquor is passed through into the evaporation zone **24** and is flash evaporated. Preferably, the die **28**, which is a device having a specific shape or design that it imparts to such material that is passed through it, is of a suitable shape or configuration that allows the preconcentrated weak black liquor to pass through the evaporation zone **24** and be

flash evaporated to form strong black liquor. Some die designs will have the added property that the preconcentrated weak black liquor will pass through the evaporation zone **24** and be flash evaporated to form strong black liquor without substantially contacting the inner surface or surfaces **32** of the evaporation zone **24**. In some operations this is desirable.

An exemplary die has a plurality of orifices or holes that allow the preconcentrated weak black liquor to form strands or streams (that may be discontinuous) as the preconcentrated weak black liquor passes through the evaporation zone **24**. Another possibility is for the die **28** to have orifices that are slits and allow for sheets or curtains (that may be discontinuous) of heated preconcentrated weak black liquor to travel or fall into the evaporation chamber **24**. These exemplary designs of the die **28** allow more surface area of preconcentrated weak black liquor to be exposed in the evaporation zone **24**, which allows for more efficient evaporation. Other configurations of the die and its orifice or orifices are also contemplated for this invention.

The at least one volatile component (volatile component being volatile at the temperature at which the preconcentrated weak black liquor enters the evaporation zone and at the pressure in the evaporation zone) that is evaporated from the preconcentrated weak black liquor to form the strong black liquor is mostly, if not all, water. Other ingredients of the preconcentrated weak black liquor may be evaporated, although that is not most desirable.

The evaporation step relies on the mechanism wherein the energy absorbed by the preconcentrated weak black liquor during the heating step is preserved by maintaining a back pressure on the preconcentrated weak black liquor to prevent vaporization of the preconcentrated weak black liquor in the inlet **26** to the evaporation zone **24**, and is subsequently released in the evaporation zone causing evaporation. The number and size of the orifices in the die **28** affect the back pressure that is maintained upstream. The back pressure must be sufficient to keep the preconcentrated weak black liquor from boiling before it enters the evaporation zone **24**.

A multi-orifice die may be preferred, particularly because the weak black liquor is to be concentrated or evaporated in a single pass through the evaporation zone. Selection

of the number and size of the orifices is used to achieve the desired pressure to prevent boiling of the preconcentrated weak black liquor prior to entering the evaporation zone.

The pressure in the evaporation zone **24** is controlled so that when the preconcentrated weak black liquor enters the evaporation zone **24** it flash evaporates. The greater the temperature differential of the preconcentrated weak black liquor, the faster will be the rate of evaporation. Flash evaporation is an elementary step in which volatile components, for example, unreacted monomers, solvents or species, are removed. In the case of weak black liquor, the change in pressure allows for flash evaporation of the volatile component(s) from the weak black liquor, resulting in concentration to form strong black liquor.

The desired level of pressure necessary in the process may be calculated using, for example, the calculations set forth in Reid et al., The Properties of Gases and Liquids, McGraw-Hill Book Co., 4<sup>th</sup> ed., Chapters 7 and 8, and Appendix A.

There may be many factors that interact in selecting the target strong black liquor concentration and the present invention can be employed to achieve whatever increased concentration (or level of evaporation) is finally selected relative to the initial concentration.

The strong black liquor will generally have a percent solids of about 45-50 wt % solids before it is fed to the recovery boiler.

### **Continuous Adjustment**

The pressure and/or temperature of the inventive process are periodically or continuously controlled and manually or automatically controlled. This is done using a plurality of temperature and pressure sensors, as described above. In addition, a conditions sensor may be used to monitor the conditions or properties of the remaining (or post-evaporation) strong black liquor. The sensors may be part of a control system(s) that can regulate the pressure and temperature of the pre-evaporation black liquor, evaporation zone, etc. In an exemplary embodiment, one or more of the variables in the inventive process (pressures, temperatures) are regulated by automatic control systems, the set points having been manually supplied by an operator. In another exemplary embodiment,

one or more of the closed-loop systems that regulate temperature and pressure will receive set points from another control system, based on directly measured or inferred properties of the post-evaporation strong black liquor. In another exemplary embodiment, the entire inventive process will be controlled by an automatic control system, based on measurements of temperature and/or pressure, and on properties of the post-evaporation strong black liquor. The operator will specify a desired property (such as percent solids content) of the post-evaporation strong black liquor, and the control system will automatically regulate process variables in such a way as to achieve the desired property.

The control system may comprise a single multivariable controller, or several independent single-loop controllers, or a combination of controller types.

### **Collection**

The concentrated black liquor is collected in a collection vessel **42**. A pump may be mounted at, in, or after the collection vessel. A pump should be selected to be of a type that can remove strong black liquor from the collection vessel and pass it at a higher pressure into an ultimate collection vessel of some type, or it could be passed to the recovery boiler (in pulp production process) from the collection vessel. This may be done continuously or periodically.



### Examples

This invention is further illustrated by the following examples, which are not intended to limit the scope of the invention. In the examples, sucrose solutions were used in place of black liquor. Black liquor is difficult to obtain for laboratory studies unless one is closely associated with a pulp manufacturing plant. It is expected that the sucrose solutions mimicked the behavior of black liquor in the inventive process. In the examples, all parts, ratios and percentages are by weight unless otherwise indicated. The following test method was used to characterize the compositions in the following examples.

### **Test Method**

#### **Solids**

Dried solids of sucrose solutions were determined from measuring the refractive index of the material and using that measurement to calculate the solids in weight percent. The refractive index of each sample was measured with a Leica Mark II Abbe Refractometer (available from Leica Microsystems, Inc., Depen, New York) with the temperature of the water bath maintained at 25°C. The concentration of each sucrose solution was calculated from the measured refractive index of the solution and a correlation between concentration and refractive index. The correlation between concentration and refractive index was determined from data in "Properties of Sucrose Solutions," *CRC Handbook of Chemistry and Physics*, 53rd Edition, page D-128, The Chemical Rubber Co., Cleveland (1972).

The following process was used to make the examples.

### Process

The material to be concentrated was placed in a pressurized, jacketed feed vessel kept at 240 kPa (20 psig) and 40°C. This material was continuously fed to the evaporation zone using the pressure in the feed tank and a valve to adjust the flow of liquid. The feed was heated by passing through a custom-made heat exchanger consisting of a jacketed tube inside an outer tube. The feed passed through the inner tube that had a length of

about 610 mm (24 in) and an outer diameter of about 6.3 mm (0.25 in). Temperature controlled water passed through the outer tube that had a diameter of about 25 mm (1 in). The material next passed through a custom-made die containing a single hole or orifice, about 1.09 mm (0.043 in) in diameter, before entering the evaporation zone. The evaporation zone consisted of a vertical portion comprising a CLAISEN™ adapter, available as Part No. 5135 from Ace Glass Inc., Vineland, New Jersey. A side arm came off the vertical portion and sloped upward to a vertical 10/30 joint, which was connected to a pressure transducer (Model number 1151AP5E333B1, available from Rosemount, Inc., Chanhassen, MN). Another side arm sloping off the side arm described immediately above had a male 24/40 fitting that was connected to a water aspirator vacuum system (custom-made). The concentrated material was continuously removed from the evaporation zone using an air-driven pump (Model FH432 from Viking Pump, Inc., Cedar Falls, Iowa).

Small jars were filled with the product leaving the evaporation zone. Each jar was filled for 1 minute and then replaced with a next one. Refractive index of the material in each of the jars was measured. The concentrations of the sucrose solutions were calculated from the measured refractive index of the solutions and the correlation between concentration and refractive index. The correlation between concentration and refractive index was determined from data presented in "Properties of Sucrose Solutions," *CRC Handbook of Chemistry and Physics*, 53rd Edition, page D-128, The Chemical Rubber Co., Cleveland (1972).

### Example 1

This Example illustrates the response time for a pressure only change.

A sucrose solution (Refractive Index = 1.4545, Concentration = 64.8 wt%) was placed in the feed tank, and flow was begun to the evaporation zone. Conditions were allowed to stabilize at a heat exchanger temperature of 71°C and an evaporation zone pressure of 9.3 kPa (70 torr). The pressure was changed and stabilized at between 5.6 and 5.7 kPa (between 42 and 43 torr). After a period of time the pressure was changed again and stabilized at between 2.8 and 2.9 kPa (21 and 22 torr).

Solids wt % was determined for all samples. Pressure, refractive index and calculated solids wt % are shown in Table 1 with elapsed time.

**Table 1**

Sample	Elapsed Time min	Pressure kPa (torr)	Refractive Index at 25°C	Wt % Solids
A	3	9.3 (70)	1.4574	66.1
B	4	Change P	1.4574	66.1
C	5	5.7 (43)	1.4592	66.8
D	6	5.7 (43)	1.4621	68.1
E	7	5.7 (43)	1.4604	67.4
F	8	5.7 (43)	1.4593	66.9
G	9	5.7 (43)	1.4607	67.5
H	10	5.7 (43)	1.4600	67.2
I	11	5.6 (42)	1.4600	67.2
J	12	5.6 (42)	1.4603	67.3
K	13	Change P	1.4605	67.4
L	14	2.9 (22)	1.4603	67.3
M	15	2.9 (22)	1.4646	69.2
N	16	2.9 (22)	1.4641	69.0
O	17	2.9 (22)	1.4639	68.9
P	18	2.8 (21)	1.4633	68.6
Q	19	2.8 (21)	1.4633	68.6
R	20	2.8 (21)	1.4631	68.5
S	21	2.8 (21)	1.4633	68.6
T	22	2.8 (21)	1.4632	68.6
U	23	2.8 (21)	1.4631	68.5

**Example 2**

This Example illustrates the response time for a temperature change only.

A sucrose solution (Refractive Index = 1.4545, Concentration = 64.8 wt%) was placed in the feed tank, and flow was begun to the evaporation zone. Conditions were allowed to stabilize at a heat exchanger temperature of 57°C and an evaporation zone pressure of between 5.1 and 5.2 kPa (between 38 and 39 torr). The temperature was changed and stabilized at 70°C. After a period of time the temperature was changed again and stabilized at 83°C.

Solids wt % was determined for all samples. Temperature, refractive index and calculated solids wt% are shown in Table 2 with elapsed time.

**Table 2**

Sample	Elapsed Time min	Temperature °C	Refractive Index at 25°C	Wt % Solids
A	11	57	1.4584	66.5
B	12	Change T	1.4584	66.5
C	13	57	1.4584	66.5
D	14	58	1.4585	66.5
E	15	63	1.4595	67.0
F	16	66	1.4607	67.5
G	17	68	1.4610	67.6
H	18	69	1.4612	67.7
I	19	69	1.4611	67.7
J	20	70	1.4615	67.8
K	21	70	1.4615	67.8
L	22	70	1.4614	67.8
M	23	70	1.4611	67.7
N	24	70	1.4609	67.6
O	25	70	1.4612	67.7
P	26	Change T	1.4611	67.7
Q	27	70	1.4618	68.0
R	28	73	1.4616	67.9
S	29	79	1.4623	68.2
T	30	81	1.4634	68.7
U	31	81	1.4641	69.0
V	32	82	1.4646	69.2
W	33	83	1.4648	69.3
X	34	83	1.4645	69.1
Y	35	82	1.4649	69.3
Z	36	84	1.4649	69.3
AA	37	84	1.4648	69.3
AB	38	82	1.4643	69.0
AC	39	82	1.4643	69.0
AD	40	83	1.4640	68.9
AE	41	83	1.4644	69.1
AF	42	83	1.4645	69.1
AG	43	83	1.4642	69.0

The easiest way to compare the dynamics of the changes in Examples 1 and 2 was to plot the normalized change in solids concentration versus the time after a deliberate change occurred in the set-point of either the pressure or the temperature. Figure 2 shows the normalized solids % changes as they relate to changes in the pressure or temperature for Examples 1 and 2 from the start of each change in condition. As seen, the response to

a change in pressure was much faster than the response to a change in temperature. One can compensate for the overshoot that occurred with a pressure change using standard methods known to those skilled in the art of process control. This would result in pressure control as a primary means of control of evaporation processes that would be much more rapid than that obtainable with temperature control alone.

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